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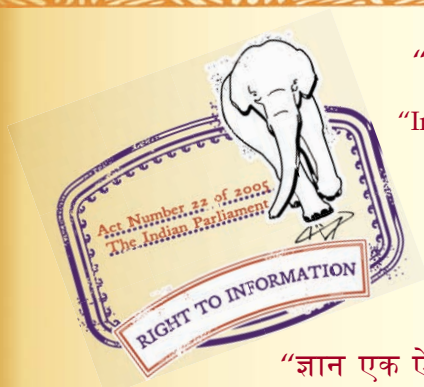
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IS 8858 (1978): Methods for spectrographic analysis of fine silver ingot (grades 9999 and 999) [MTD 10: Precious Metals]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

METHOD FOR SPECTROGRAPHIC ANALYSIS OF FINE SILVER INGOT (GRADES 9999 AND 999)

UDC 669.22.412 : 543.42



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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Price Rs 5.00

Gr 2

December 1978

Indian Standard

METHOD FOR SPECTROGRAPHIC ANALYSIS OF FINE SILVER INGOT (GRADES 9999 AND 999)

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Indian Standard

METHOD FOR SPECTROGRAPHIC ANALYSIS OF FINE SILVER INGOT (GRADES 9999 AND 999)

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 26 July 1978, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Three grades of fine silver are specified in IS : 5320-1969*. Need was felt for a standard which should lay down spectrographic methods for determining elements prescribed for Grades 9999 and 999.

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

1. SCOPE

1.1 This standard prescribes spectrographic method for the determination of metallic impurities for grades 9999 and 999 silver as specified in IS : 5320-1969*. However, the methods do not cover Pt, Pd, Se and Te for grade 9999.

2. METHOD OF DETERMINATION

2.0 Silver is converted to silver chloride and analysed spectrographically.

2.1 Standards

2.1.1 A set of standards is prepared on silver chloride matrix to cover the concentration range of 0.1 ppm to 25 ppm. A master standard of 1 000 ppm is prepared by dry grinding specpure silver chloride with the synthetic mixture of compounds of the required impurities. Standards of lower concentrations are prepared by successive dilutions with specpure silver chloride. All the standards are mixed with specpure graphite in the ratio of 1 : 1 and ground thoroughly in an agate mortar.

*Specification for fine silver ingot.

†Rules for rounding off numerical values (revised).

2.1.2 Preparation of Samples — The sample of metallic silver is dissolved in the minimum amount of high purity concentrated nitric acid and evaporated to dryness. The silver nitrate is dissolved in redistilled water and heated to boiling. High purity dilute hydrochloric acid (1 : 1) is then added with constant stirring to precipitate the silver completely as silver chloride. Silver Chloride along with the supernatant liquid, is dried on a water-bath and ground in an agate mortar with high purity graphite powder in the ratio of 1 : 1.

2.2 Excitation and Photography of the Spectra

2.2.1 Electrode System — The lower electrode is a 6-mm diameter high purity graphite rod having a cup cut at one end with a 5 mm inside diameter and a depth of 3 mm. Silver chloride graphite mixture is packed into this crater. The upper electrode (cathode) is a 6-mm diameter high purity graphite rod pointed to a 30° cone. The gap between the electrodes is initially set at 4 mm.

2.2.2 Spectrograph — Any commercial grating or quartz prism spectrograph having a linear dispersion of at least 0.7 nm/mm at 350 nm may be used.

2.2.3 Excitation — Anode excitation is employed to excite samples and standards in the dc arc at a current of 10 A by short-circuiting the gap between anode and cathode by a pure graphite rod.

2.2.4 Exposure Conditions

Wave length region	232-358 nm
Slit width	0.010 mm
Slit length	2 mm
Diaphragm	A 4-mm aperture at collimating lens or mirror to cut off radiation from glowing electrode tips
Exposure	20 s
Charge	40 mg

NOTE — For grade 999, a filter or step sector giving 10 percent transmission should be used.

2.2.5 Photographic Plates/Films — The spectra are to be recorded on photographic emulsion suitable for the spectral region.

2.2.6 Photographic Processing — The plates are developed in accordance with IS : 6010-1971*.

*Recommended practice for photographic processing in spectrochemical analysis.

2.2.7 Plate Calibration — Emulsion calibration is done by employing the step sector method as described in Draft Indian Standard recommended practice for calibration of emulsion for photographic photometry (*under preparation*).

NOTE — Until this standard is published this may be done as agreed to between the parties.

2.2.8 Line Pairs for Microphotometer Measurement — The analytical line pairs shall be as follows:

<i>Analytical Line</i>	<i>Internal Standard</i>
nm	nm
Co 242·49	Ag 247·38
B 249·77	„
Sb 259·80	„
Fe 259·84	„
Mn 260·57	„
Mg 277·93	„
Pb 283·30	„
Cr 283·56	„
Sn 283·99	„
Ca 294·36	Ag 309·91
Ni 305·08	„
Bi 306·77	„
Al 303·21	„
Cu 324·75	„
In 325·60	„

NOTE — For Grade 999 exposure.

2.2.9 Analytical Curve — Spectra of the standards and sample shall be photographed in duplicate on each plate. The densities of the lines as recommended under 2.2.8 shall be measured by a microphotometer and converted into line intensities after background correction. The average of log intensity ratio of analytical line to internal standard lines is plotted against log concentration of the element. Sample intensity ratio values are read on the curve to obtain the concentration.

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